R. F. Mather

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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

Quarterly Progress Report 2

EDITED BY E. E. HOFFMAN

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
Under Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI 15, OHIO

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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

QUARTERLY PROGRESS REPORT 2

Covering the Period
October 15, 1963 through January 15, 1964

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

I INTRODUCTION

This report covers the period, from October 15, 1963 to January 15, 1964, of a program to develop a prototype corrosion test loop for the evaluation of refractory alloys in boiling and condensing potassium environments which simulate projected space electric power systems. The envisioned prototype test consists of a two-loop Cb-1Zr facility; sodium will be heated by direct resistance in a primary loop and will be used in a heat exchanger to boil potassium in the secondary, corrosion test loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high vacuum environment. The immediate corrosion test design conditions are shown below; it is expected that the temperatures could be increased by about 400°F when testing is extended to include refractory alloys stronger than Cb-1Zr.

1. Boiling temperature, 1900°F
2. Superheat temperature, 2000°F
3. Condensing temperature, 1350°F
4. Subcooling temperature, 800°F
5. Mass flow rate, 20 to 40 lb/hr
6. Vapor velocity, 100 to 150 ft/sec
7. Average heat flux in the potassium boiler - 50,000 to 100,000 BTU/hr ft^2

The development program will proceed with the construction and operation of three Cb-1Zr test loops, each of which will be used in a sequence of component evaluation and endurance testing. Loop I, a natural convection loop, will be operated for 1,000 hours with liquid sodium at 2200°F to evaluate the electrical power vacuum feed-throughs, thermocouples, the method of attaching the electrodes, the electrical resistivity characteristics of the heater segment, and the use of thermal and electrical insulation. Loop II, a single-phase, forced-circulation loop, will be operated for 2,500 hours with liquid sodium at about 2100°F to evaluate the primary loop EM pump, a flowmeter, flow control and isolation valves, and pressure transducers. The Pre-prototype Corrosion Test Loop, a two-loop system, will include a boiler, turbine simulator, and condenser in addition to the above components. This loop facility will be used to develop and endurance test (2,500 hours) the components required to achieve stable operation at the corrosion test design conditions.

The quarterly reports issued for this program will summarize the status of the work with respect to design considerations, construction procedures, and test results. Detailed topical reports will also be issued to describe each test loop. Additional topical reports will be prepared to cover such areas as materials specifications, purification of potassium and sodium, and inert gas purification and analysis.

II PROGRAM STATUS

1. Loop Design

The design of Component Evaluation Test Loop II, the single-phase, forced-circulation loop in which sodium will be circulated at 2100°F, has been essentially completed. Figure 1 is an isometric drawing of Loop II and indicates the orientation of the principal loop components. The preliminary performance test conducted on the vacuum environmental chamber at manufacturer's plant will be discussed later in this report. All loop components which will come in contact with the sodium are constructed of Cb-1Zr alloy with the exception of the molybdenum alloy (TZM) valve plug and pin. The valve temperature will be maintained below 800°F. A fast response pressure transducer designed so that only refractory alloys are in contact with the alkali metal will be put into Loop II if available prior to the filling of the loop with sodium.

The design of the Pre-prototype Corrosion Test Loop, a two loop system, has been continued during this quarter. Design selections have been made for all of the principal loop components and detail design of the system has begun. A radiation type of preheater will be used to heat the liquid potassium to the saturation temperature. The tube-in-tube, helical, sodium-to-potassium boiler will include a wire-wrapped rod type of insert in the entrance region of the boiler. The turbine simulator will consist of 10 pairs of nozzles and blades in series, sized to give a vapor velocity of 1,000 feet per second in the nozzle throat and a quality of 88%. Potassium flow in this system will be approximately 40 pounds per hour.

2. Material and Equipment Procurement

Most of the Cb-1Zr alloy, with the exception of the 200 feet of 3/8-inch OD tubing, which will be used in the construction of Loop II and the Pre-prototype Loop has been received. The inspection of Cb-1Zr alloy is proceeding and, in general, the results obtained have satisfied the materials specification requirements of the program.

The environmental test chamber for Loop II, which has inside working dimensions of 54 inches in height x 24 inches in diameter, was received and installed at General Electric in mid-January. This equipment is shown in Figure 2. Prior to shipment, the system successfully passed the preliminary acceptance test at the manufacturer's plant. A pressure of 4.1×10^{-10} torr was achieved in less than 24 hours following a 30-minute exposure to air. This pressure was obtained without the use of the titanium sublimation pumps which are to be used to handle peak outgassing loads.

The construction of the 128-inch high x 48-inch diameter vacuum chamber for the Pre-prototype Loop is nearly complete, and the system should be shipped to General Electric in February.

3. Component Evaluation Test Loop I

The construction, filling, and installation of Loop I was completed during the last quarter. A number of the Loop I components are shown in Figure 3. Prior to welding the loop and annealing these weldments, equipment qualification tests were conducted with Cb-1Zr alloy specimens. All weld specimens passed the requirements of specification SPPS-3B entitled, "Welding of Columbium - 1% Zirconium Alloy by the Inert-Gas Tungsten Arc Process."

The fabricated loop is shown in Figure 4. Following fabrication, the loop was helium leak checked and baked out at 250°F prior to filling the loop with 112 grams of purified sodium. An in-line sampler was used to retain a portion of the hot-trapped sodium during the filling of the loop. The sodium was found to contain 20 ppm oxygen as determined by seven analyses using the mercury amalgamation procedure. The standard deviation for this method is $\pm 2.5\,\mu$ gm oxygen as determined in this laboratory on potassium. Insufficient data are available at this time to establish the standard deviation for sodium.

Loop I was then installed in the vacuum test chamber which was shown in the first progress report on this program(1). The instrumented and insulated loop is shown mounted in the test chamber in Figure 5, and the associated equipment, including the partial pressure analyzer, power supply, temperature controller, and recording instruments, are shown in Figure 6.

4. Loop II Fabrication

Prior to the welding of Loop II components, a preliminary qualification test of the welding equipment was conducted according to requirements defined in Specification SPPS-3C. This specification requires that the inert gas be purified to contain less than 1 ppm active impurities by volume and that the contamination of the weld metal be held to less than 50 ppm oxygen, 50 ppm nitrogen, 10 ppm carbon, and 5 ppm hydrogen. (The mass spectrometer, helium analysis system which will eventually be used to monitor the gas purity is not yet in operation, and its status will be discussed in more detail below. Preliminary monitoring of the purified helium used to back-fill the chamber is now being conducted with the Brady apparatus to determine the oxygen concentration and

(1) Potassium Corrosion Test Loop Development, Quarterly Progress Report 1, Covering the Period July 15, 1963 through October 15, 1963, NASA Contract NAS3-2547.

a dewpoint cup to determine the moisture concentration.) For the qualification test, the chamber was evacuated to less than 1×10^{-5} torr and had a leak rate of 2.3 microns per hour prior to filling with helium (99.99% purity) which was passed through the Linde Type 13X molecular sieve. Hot trapping and analysis of the helium was not conducted for this equipment qualification test. The results of chemical analyses of the weld metal and post-weld annealed specimens satisfied the requirements of Specification SPPS-3C and are compared with the base material and filler wire in Table I.

The welding chamber in which these tests were conducted and in which Loop II and the Pre-prototype Loop will be constructed is shown in Figures 7 and 8. No field welds will be used in the fabrication of Loop II and the Pre-prototype Loop I. The large welding tank volume required to accommodate the loops during final assembly and subsequent modification, if required, is provided by the extension tank shown in Figure 8.

The first weld on the Cb-1Zr EM pump for Loop II was made in December. Control weld bend specimens were prepared prior to and following welding the pump components as required by the welding specification.

The Cb-1Zr bellows to be used in the construction of the isolation and control valves of Loop II and the Pre-prototype Loop have been fabricated. The bellows were fabricated by a hydraulic process by Standard-Thomson Corporation and although some failures occurred, 35 bellows of 71 attempted were found to be helium leak tight by the vendor. Additional quality assurance tests will be conducted on the bellows before they are shipped to Hoke, Inc., the valve manufacturer.

The machining of the pressure transducer components by the Taylor Instrument Company has been completed and the parts have been shipped. The welding of the pressure transducer parts constructed of Cb-1Zr alloy as well as all other Cb-1Zr loop components will be performed in the welding chamber described above or by electron beam welding at General Electric.

5. Helium Purification System

During the past quarter the helium purification system was completed, attached to the welding chamber, and used successfully in the welding of the EM pump as noted above. The temporary system used to purify, cool, and analyze the helium is shown in Figure 9. The gas cooling capability was found to be adequate at flow rates up to 750 SCFH. During the backfills for welding of the EM pump, the titanium hot trap was at 1400° - 1450°F and the flow rate used was about 200 SCFH. Both oxygen and water were measured at less than 0.6 ppm in the helium used to fill the welding chamber.

TABLE I

RESULTS OF PRELIMINARY WELDING AND ANNEALING QUALIFICATION TESTS* WITH THE Cb-1Zr ALLOY

	Condition	Concentration (ppm) O	N	H	C
1.	Base metal analysis by vendor	130	85	1	50
2.	Base metal analysis by General Electric	142	22	2	30, 40
3.	Filler wire	59	25	1	40
4.	Weld metal of this Qualification Test	106, 112	34, 41	2, 3	40
5.	Base metal heat treated 1 hour at 2200°F in vacuum	138	44	2	10, 20

* Welding and annealing in accordance to Specification SPPS-3C with the exception of the helium purification and analysis procedure.

During one of the preliminary gas filling operations, it was discovered that the titanium hot trap was producing "smoke" which was subsequently identified as primarily magnesium originating from the titanium sponge which contained up to 0.4% magnesium. This "smoke" has been eliminated by replacing the titanium sponge with titanium turnings.

6. Helium Analysis System

The mass spectrometer, helium analysis system has been completely assembled. On initial pump-down, a vacuum of 5×10^{-8} torr was achieved after three days of pumping on the unbaked system with only the glass trap cooled with liquid nitrogen. A thorough bakeout of the system is in progress.

Figure 10 is a photograph of the partially assembled sampling and analysis system. In the foreground are the mass spectrometer analyzer tube and magnet. A schematic diagram of the system was shown previously[1]. Figure 11 shows the completely assembled system with the bakeout oven in place, enclosing the components which are shown in Figure 10. The glass cold trap is immersed in a Dewar flask containing liquid nitrogen. Above this is the metal cold trap which, is enclosed by the heating mantle in the photograph. The mercury diffusion pump and foreline system are also visible behind the cold trap. Controls for the partial pressure analyzer, ionization gauge, thermocouple gauge, and pump heater are located on the instrument cart shown in Figure 11. During the bakeout procedure, the recorder monitors the temperature indicated by five thermocouples attached to various components.

The bakeout procedure will be completed in the near future, and the mass spectrum of the residual gases in the system will be investigated. The system should then be ready for analysis of the welding chamber gases.

---

(1) Potassium Corrosion Test Loop Development, Quarterly Progress Report 1, Covering the Period July 15, 1963 through October 15, 1963, NASA Contract NAS3-2547.

III FUTURE WORK

The detailed drawings, filling procedure, and test plan for Component Evaluation Test Loop II will be submitted to NASA for approval. The detailing of Pre-prototype Loop components will continue.

A final acceptance test will be conducted at General Electric on the 24-inch diameter x 54-inch high vacuum test chamber. The 48-inch diameter x 128-inch high environmental test facility for the Pre-prototype Loop will be installed and tested.

Test Loop I will be placed in operation following baking of the vacuum test chamber. A partial pressure analyzer will be used to monitor the concentration of residual gases in the test environment.

The fabrication of Loop II will proceed rapidly during the next quarter. The construction of the Hoke valves, the EM pump and the Taylor Instrument Company pressure transducers will be completed during this period.

The bakeout procedure for the helium analysis system will be completed and the mass spectrum of the residual gases will be investigated. The system will then be ready for analysis of the welding chamber gas.

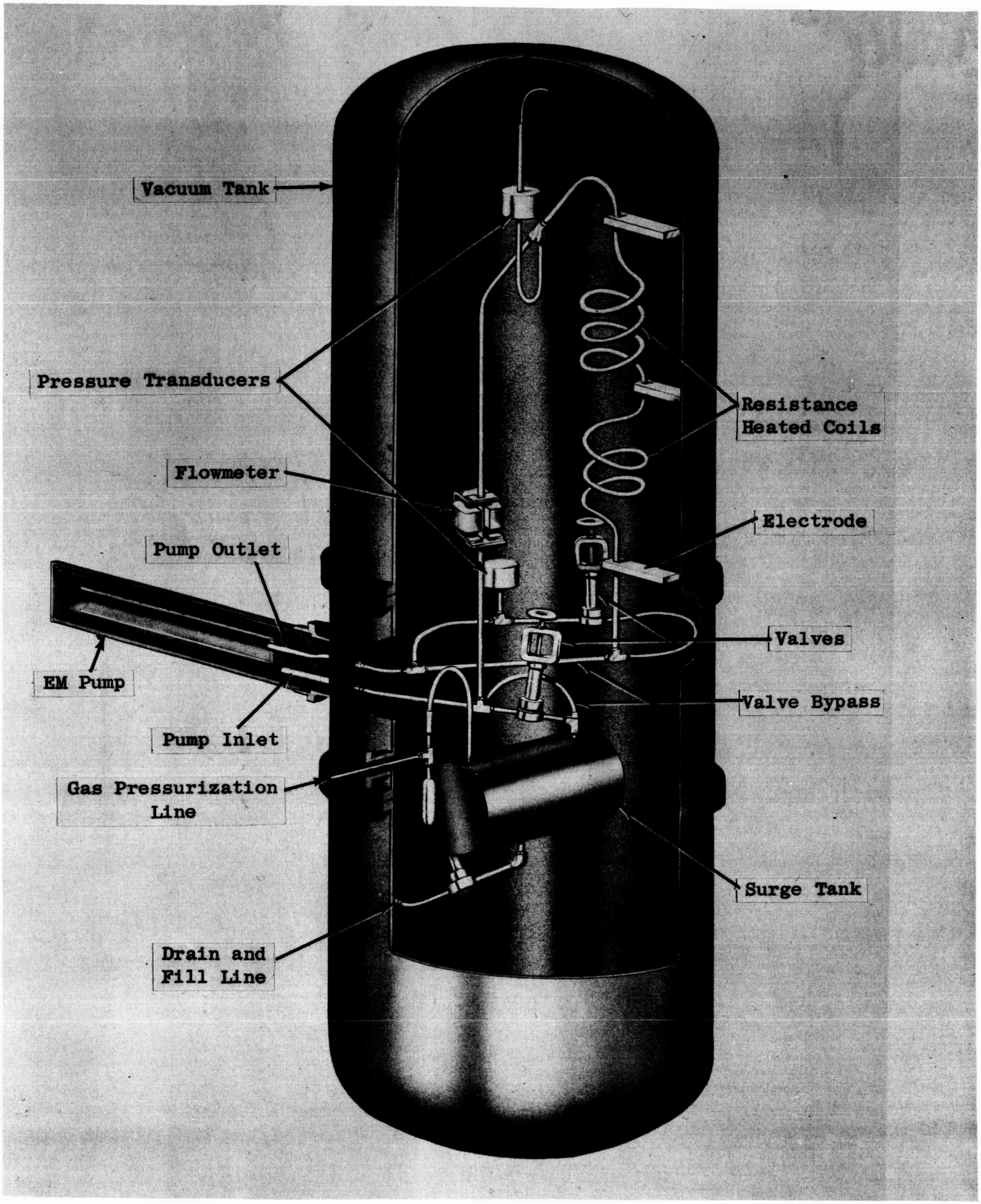

Figure 1. Isometric of Component Evaluation Test Loop II. (C64021610)

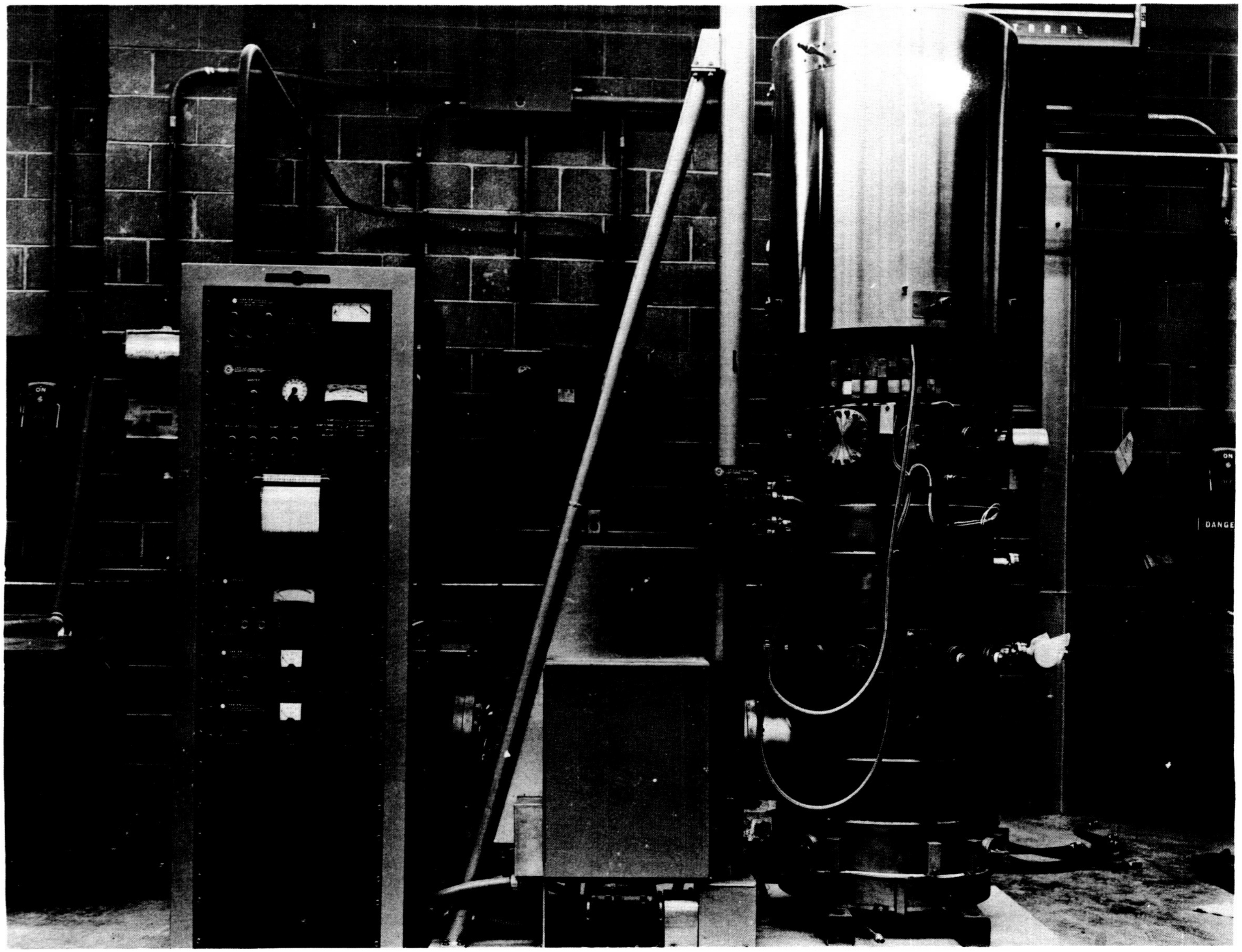

Figure 2. High Vacuum System (10^{-10} Torr Range) Used with Component Evaluation Test Loop II. The Chamber is 24 Inches in Diameter and 54 Inches High and Incorporates a 1,000 l/sec Getter-Ion Pump and a 10,000 l/sec Titanium Sublimation Pump. A 22-Inch High Spool Piece for Supporting the Loop is Shown in Place Between the Upper and Lower Portions of the Vacuum Chamber. (C64011410)

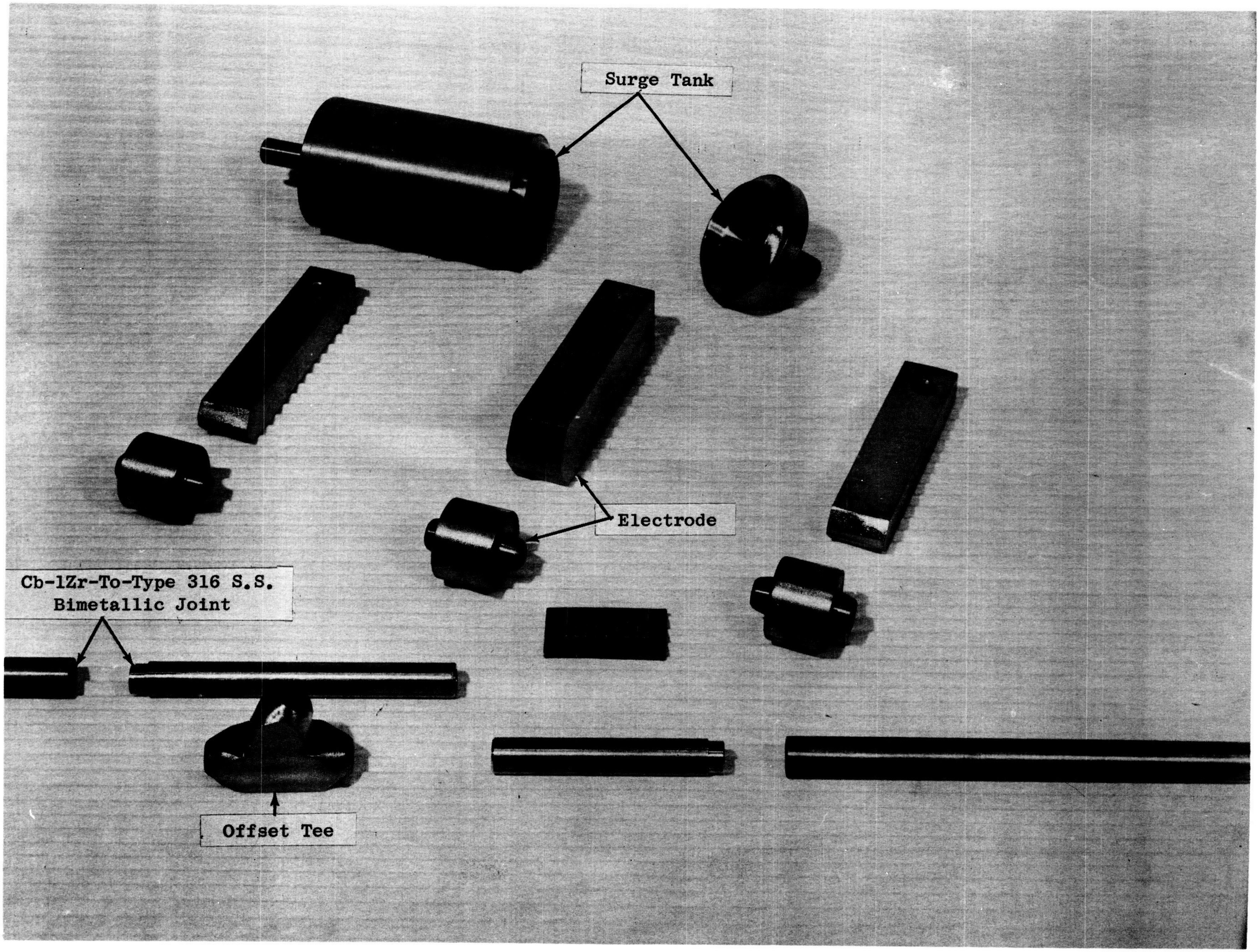

Figure 3. Component Evaluation Test Loop I Components. (C63102205)

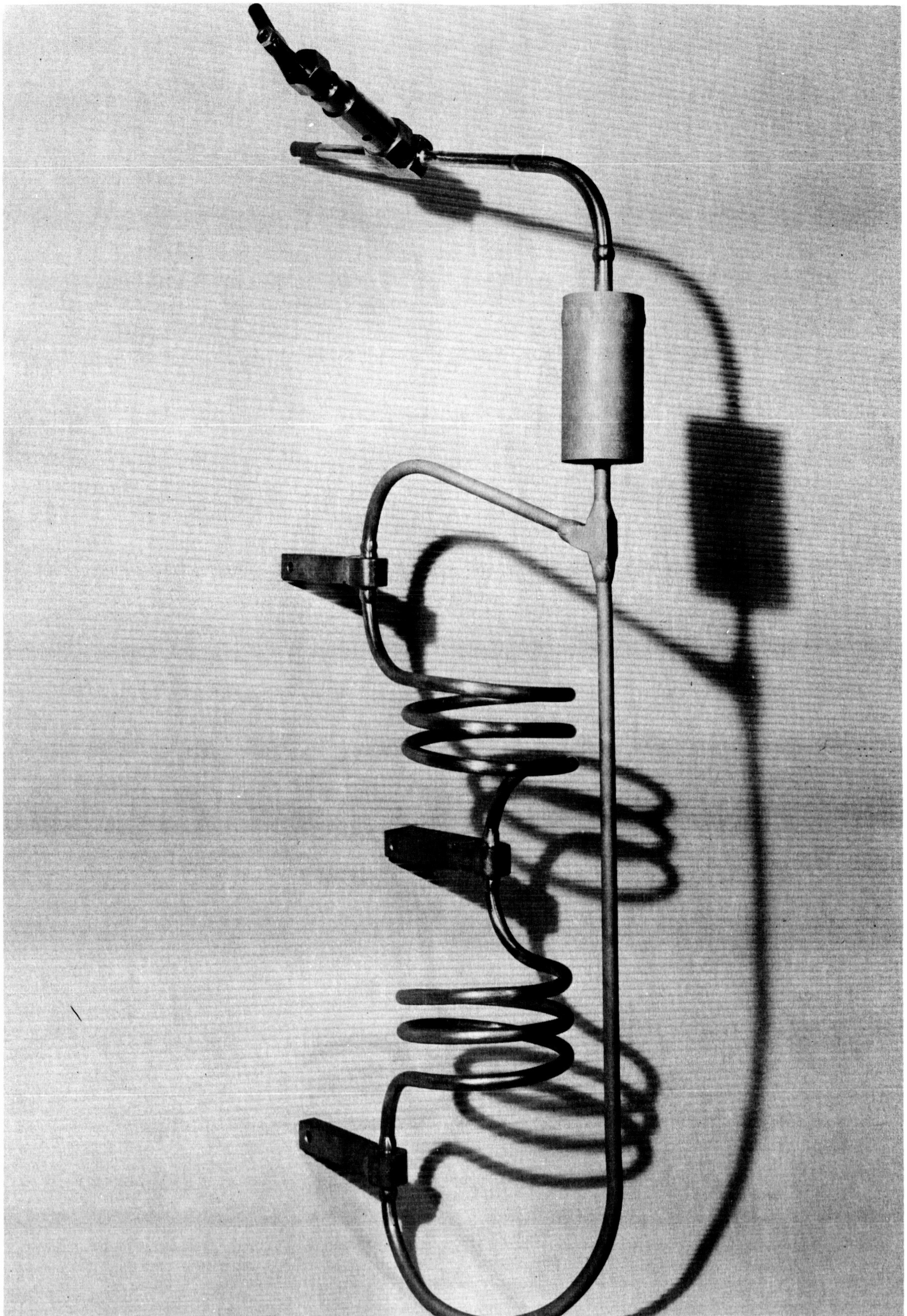

Figure 4. Component Evaluation Test Loop I. (C63121903)

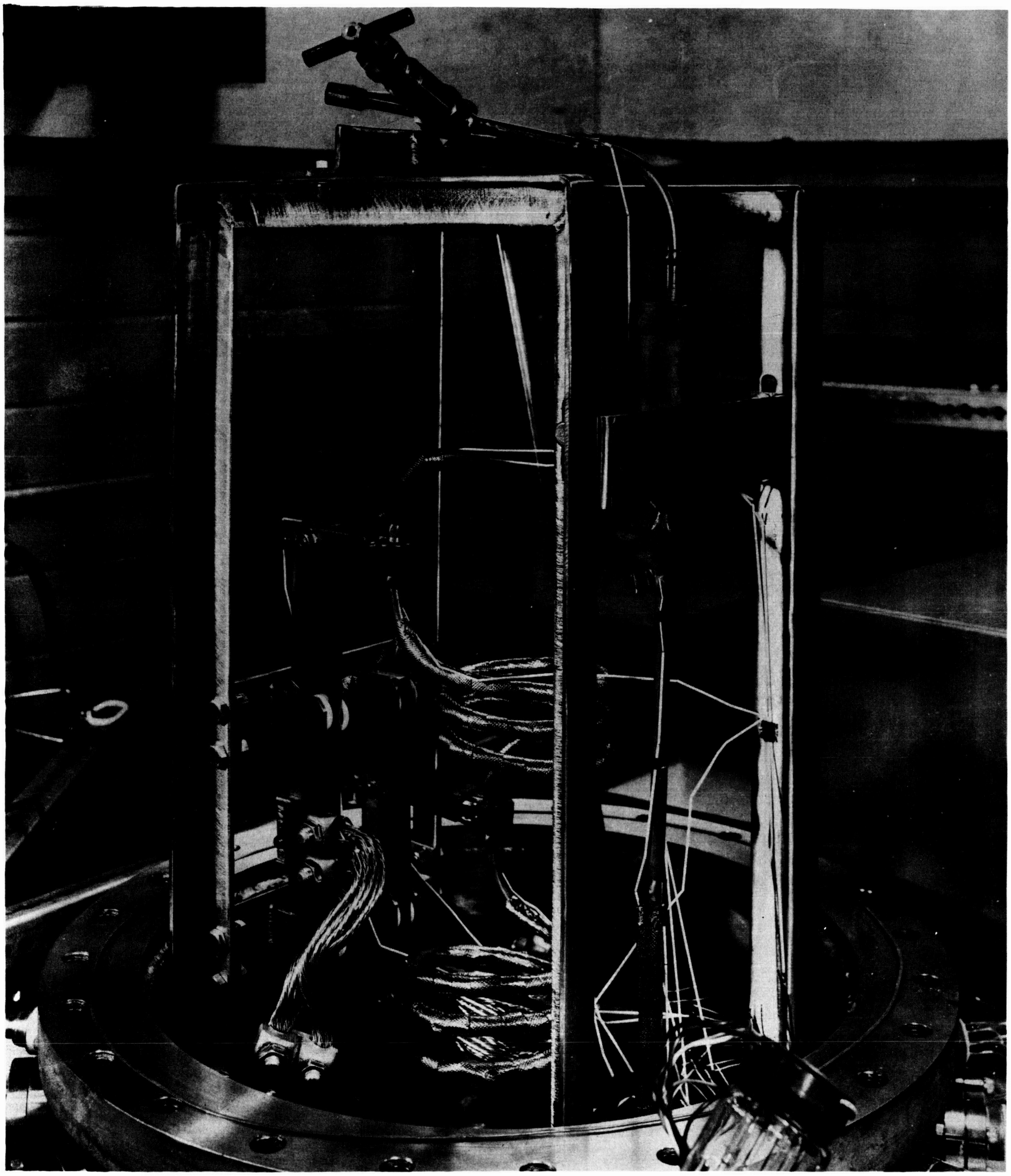

Figure 5. Component Evaluation Test Loop I Installed in Vacuum Test Chamber. (C64012330)

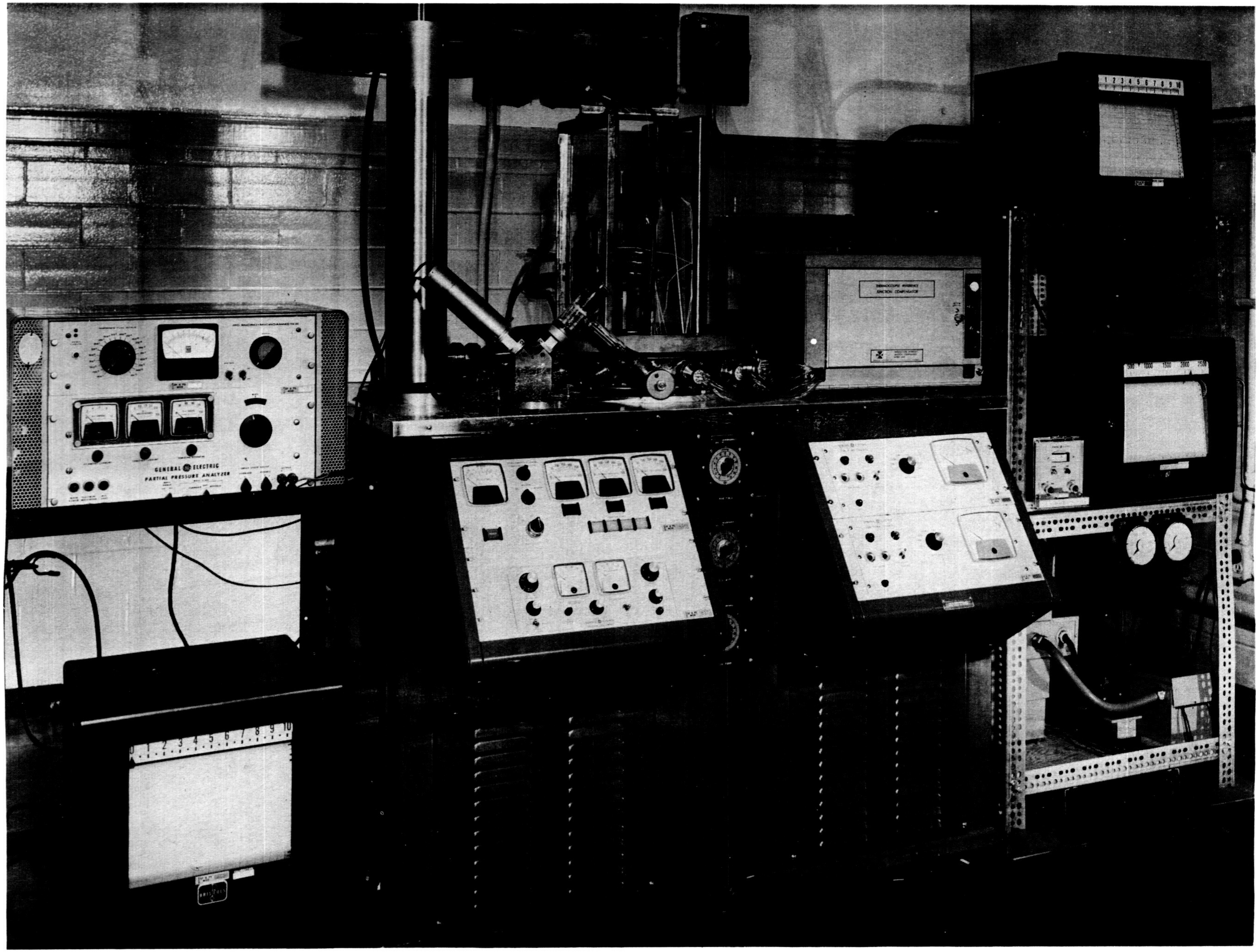

Figure 6. Component Evaluation Test Loop I and Associated Equipment. (C64012331)

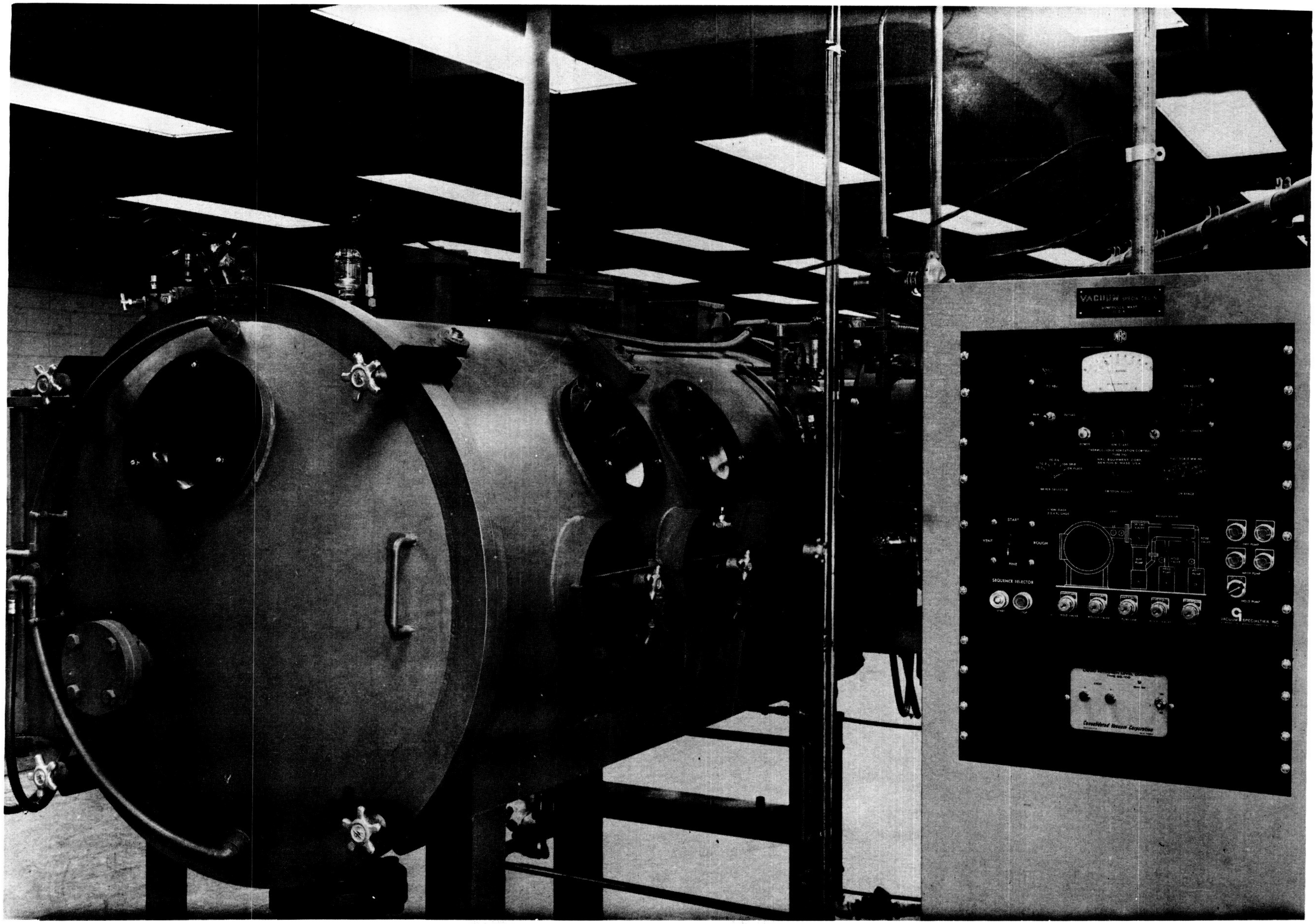

Figure 7. Vacuum-Purge, Controlled Atmosphere Welding Chamber, 3 Feet in Diameter x 6 Feet Long, Equipped with a 10-Inch Diameter Oil Diffusion Pump Capable of Attaining a Pumping Speed of 4100 l/sec at a Pressure of 1×10^{-5} Torr. (C63120630)

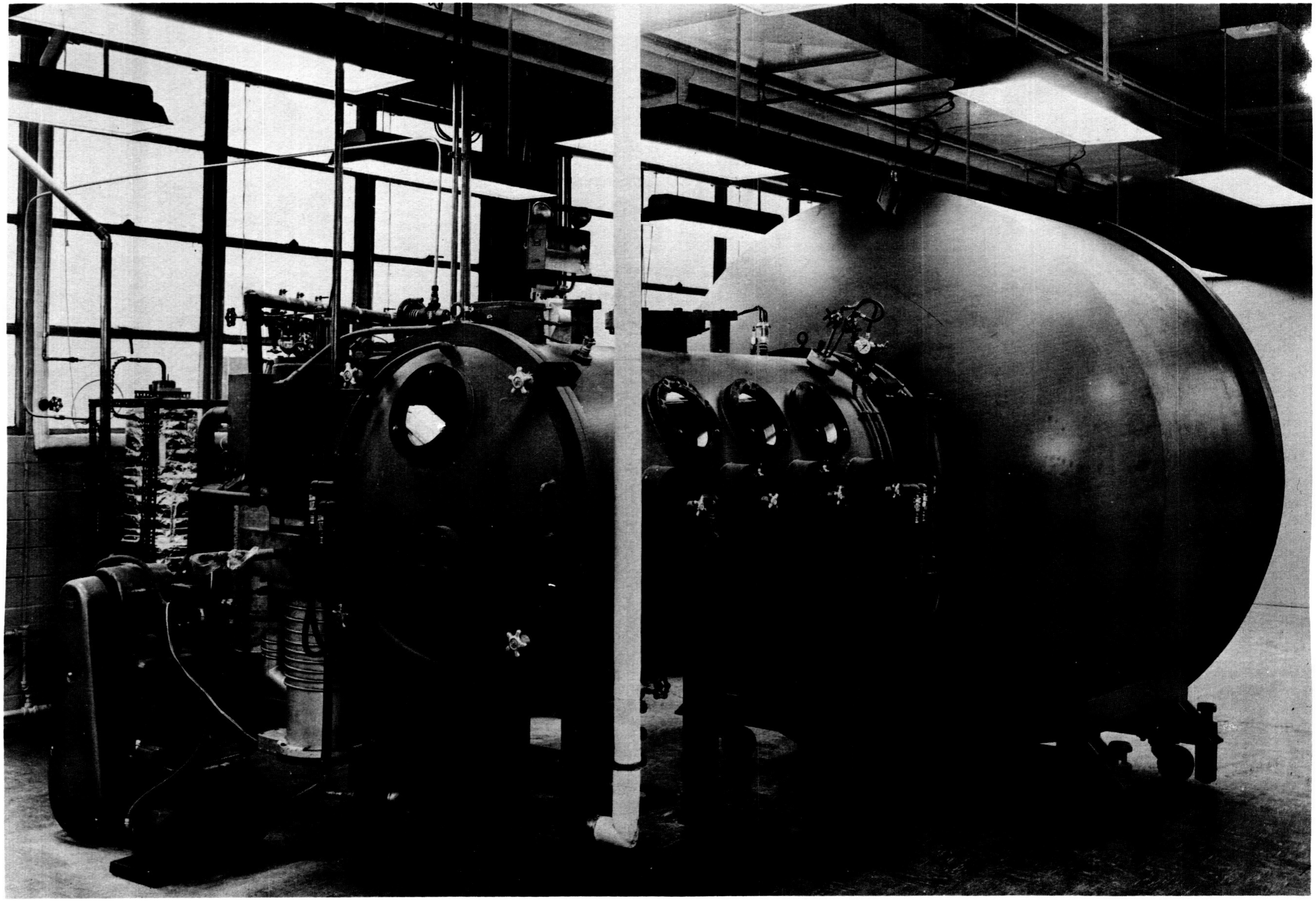

Figure 8. Vacuum-Purge, Controlled Atmosphere Welding Chamber, 3 Feet in Diameter x 6 Feet Long, Equipped with a 10-Inch Diameter Oil Diffusion Pump Capable of Attaining a Pumping Speed of 4100 l/sec at a Pressure of 1 x 10^{-5} Torr. An Extension to the Chamber, 8 Feet in Diameter x 6 Feet Long, is Shown in Place. (C63120631)

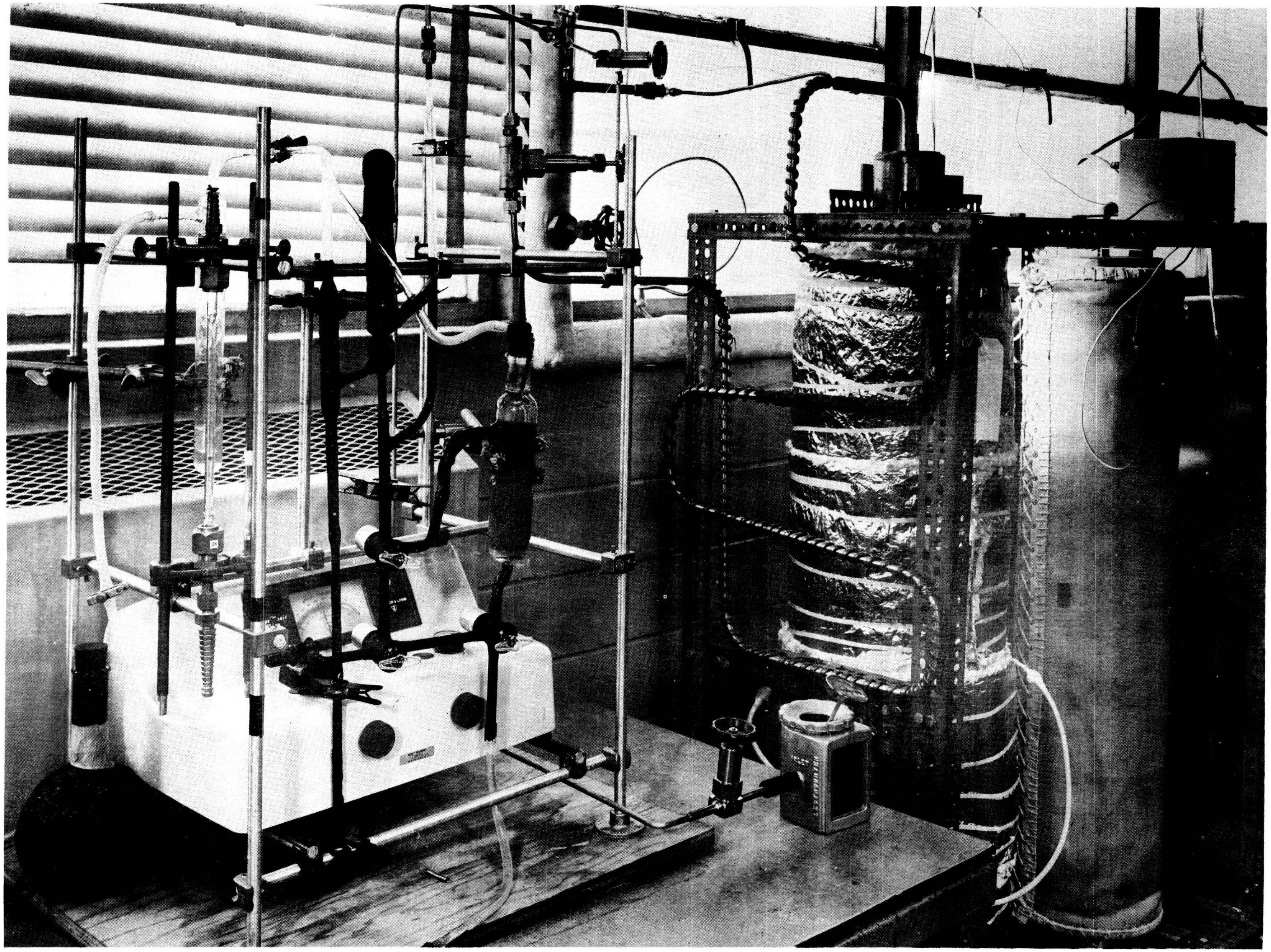

Figure 9. Temporary Helium Gas Purification Train and Cooler with Brady Apparatus and Dewpoint Cup Attached. (C63122030)

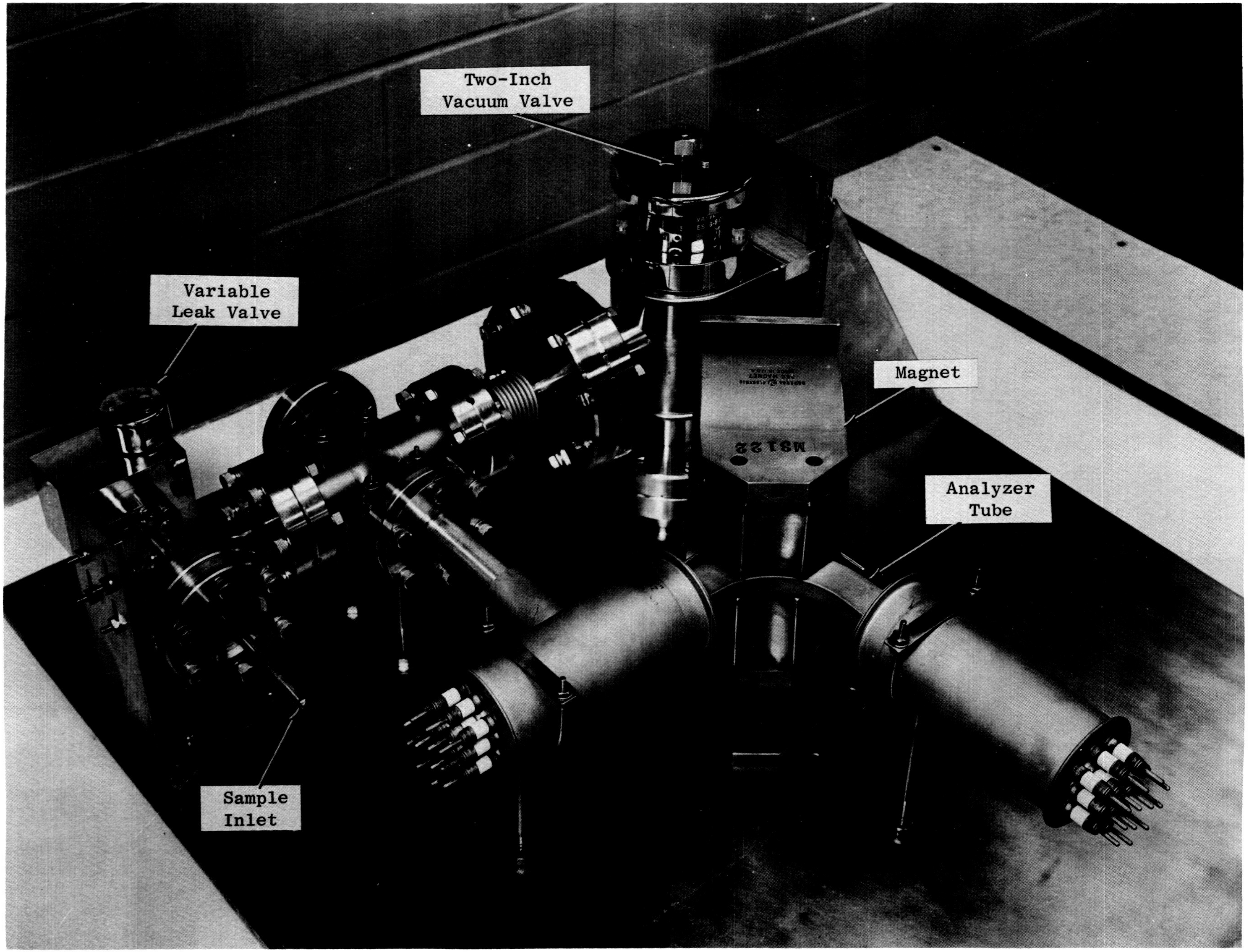

Figure 10. Helium Analysis System Partially Assembled. (C63122028)

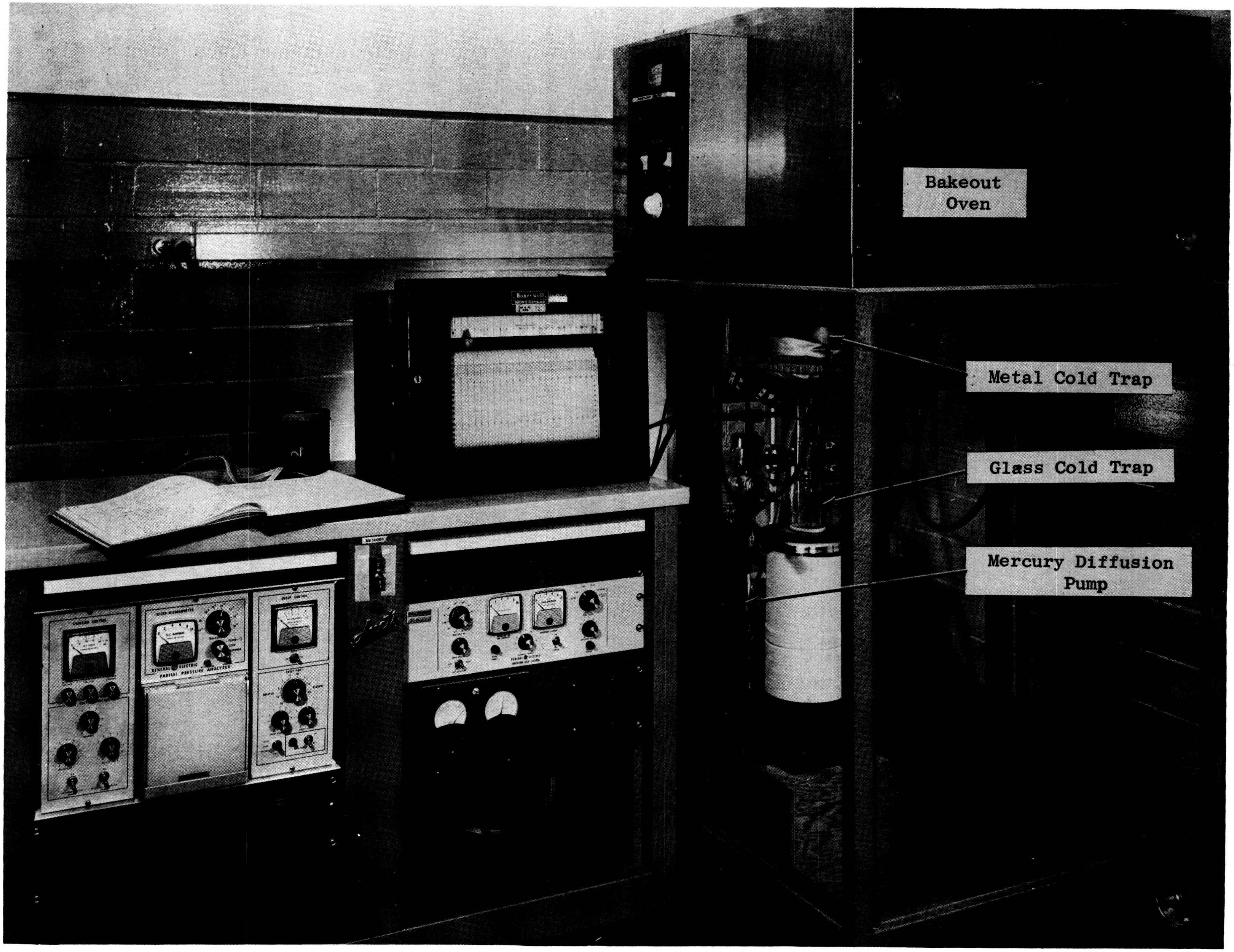

Figure 11. Helium Analysis System with the Bakeout Oven in Place.
(C64011609)

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Westinghouse Electric Corporation
Aerospace Electrical Division
Wapak Road
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Attention: Paul Kueser

Wolverine Tube Division
Calumet and Hecla Incorporated
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American Machine and Foundry Company
Alexandria Division
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Douglas Aircraft Company, Inc.
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